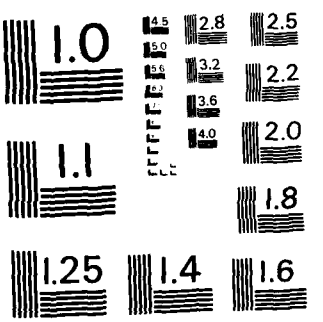
AD-A142 425 TRACE ANALYSIS IN QUARTZ(U) EAGLE-PICHER INDUSTRIES INC 1/1
MIAMI OK A L FLUESMEIER MAR 84 RADC-TR-84-51
F19628-81-C-0072

UNCLASSIFIED F/G 7/4 NL

END
DATE FILMED
8 84
DTIC



(12)

RADC-TR-84-51
Final Technical Report
March 1984

# TRACE ANALYSIS IN QUARTZ

Eagle-Picher Industries, Inc.

A. L. Fluesmeier



APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED



ROME AIR DEVELOPMENT CENTER
Air Force Systems Command
Griffiss Air Force Base, NY 13441

This report has been reviewed by the RADC Public Affairs Office (PA) and is releasable to the National Technical Information Service (NTIS). At NTIS it will be releasable to the general public, including foreign nations.

RADC-TR-84-51 has been reviewed and is approved for publication.

APPROVED: Alton J. Armington

ALTON F. ARMINGTON
Project Engineer

APPROVED: Harold Roth

HAROLD ROTH, Director
Solid State Sciences Division

FOR THE COMMANDER: John A. Ritz

JOHN A. RITZ
Acting Chief, Plans Office

If your address has changed or if you wish to be removed from the RADC mailing list, or if the addressee is no longer employed by your organization, please notify RADC (ESME) Hanscom AFB MA 01731. This will assist us in maintaining a current mailing list.

Do not return copies of this report unless contractual obligations or notices on a specific document requires that it be returned.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE

## REPORT DOCUMENTATION PAGE

| | |
|---|---|
| 1a. REPORT SECURITY CLASSIFICATION<br>UNCLASSIFIED | 1b. RESTRICTIVE MARKINGS<br>N/A |
| 2a. SECURITY CLASSIFICATION AUTHORITY<br>N/A | 3. DISTRIBUTION/AVAILABILITY OF REPORT<br>Approved for public release; distribution unlimited |
| 2b. DECLASSIFICATION/DOWNGRADING SCHEDULE<br>N/A | |
| 4. PERFORMING ORGANIZATION REPORT NUMBER(S)<br>N/A | 5. MONITORING ORGANIZATION REPORT NUMBER(S)<br>RADC-TR-84-51 |
| 6a. NAME OF PERFORMING ORGANIZATION<br>Eagle-Picher Industries, Inc.<br>Specialty Metals Division<br>6b. OFFICE SYMBOL (If applicable) | 7a. NAME OF MONITORING ORGANIZATION<br>Rome Air Development Center (ESME) |
| 6c. ADDRESS (City, State and ZIP Code)<br>P.O. Box 1090<br>Miami OK 74354 | 7b. ADDRESS (City, State and ZIP Code)<br>Hanscom AFB MA 01731 |
| 8a. NAME OF FUNDING/SPONSORING ORGANIZATION<br>Rome Air Development Center<br>8b. OFFICE SYMBOL (If applicable)<br>ESME | 9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER<br>F19628-81-C-0072 |
| 8c. ADDRESS (City, State and ZIP Code)<br>Hanscom AFB MA 01731 | 10. SOURCE OF FUNDING NOS.<br>PROGRAM ELEMENT NO. 62702F; PROJECT NO. 4600; TASK NO. 17; WORK UNIT NO. 49 |
| 11. TITLE (Include Security Classification)<br>TRACE ANALYSIS IN QUARTZ | |
| 12. PERSONAL AUTHOR(S)<br>A. L. Fluesmeier | |
| 13a. TYPE OF REPORT<br>Final<br>13b. TIME COVERED<br>FROM Jul 81 TO Dec 83 | 14. DATE OF REPORT (Yr., Mo., Day)<br>March 1984<br>15. PAGE COUNT<br>24 |
| 16. SUPPLEMENTARY NOTATION<br>N/A | |

| 17. COSATI CODES | | | 18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number) |
|---|---|---|---|
| FIELD | GROUP | SUB. GR. | Quartz Crystals<br>Chemical Analysis<br>Atomic Absorption |
| 07 | 02 | | |
| | | | |

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

The objective of this work was to devise a technique capable of determining trace impurities in quartz at the parts per million range. The technique selected was atomic absorption and a procedure was devised for determining lithium, sodium, potassium, iron, aluminum and carbon. Over 150 quartz samples were analyzed using this technique.

| | |
|---|---|
| 20. DISTRIBUTION/AVAILABILITY OF ABSTRACT<br>UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT. ☐ DTIC USERS ☐ | 21. ABSTRACT SECURITY CLASSIFICATION<br>UNCLASSIFIED |
| 22a. NAME OF RESPONSIBLE INDIVIDUAL<br>Alton F. Armington | 22b. TELEPHONE NUMBER (Include Area Code)<br>(617) 861-4834<br>22c. OFFICE SYMBOL<br>RADC (ESME) |

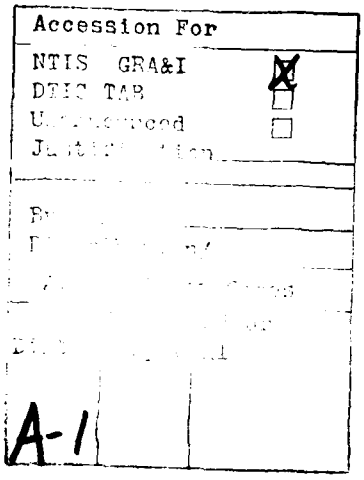
DD FORM 1473, 83 APR EDITION OF 1 JAN 73 IS OBSOLETE.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE

TABLE OF CONTENTS






This final report covers the work performed on Department of the Air Force Contract No. F19628-81-C-0072 by the Analytical Department of Eagle-Picher Industries Inc.,Miami, Oklahoma and covers a period of time from July 1, 1981 through December 31, 1983.

## I LITERATURE SURVEY

The first part of this contract was the performance of a literature survey of analytical methods for trace analysis in quartz and fluoride glasses.

During this search the Dialog on-line retrieval service was used. This service allows many sources to be checked in a relatively short time.

The literature for the period 1972-1981 inclusive dealing with quartz and fluoride glasses was surveyed using the Chemical Abstracts data base. Key words which were used in the broadly defined search were the following: quartz, impurity, silica, lithium, sodium, potassium, aluminum, fluoride, fluoride glass, analysis, determination and amorphous. A total of more than 2100 articles were identified about 10% of which were pertinent to the project. It is not surprising that most of the analytical references found were for impurity levels much higher than we were interested in. It therefore became necessary to develop an analytical procedure suitable to our purposes. The results of the literature search were forwarded to the

contract monitor. The analytical procedure developed in this laboratory was used for subsequent analytical work on this contract. A copy of this procedure can be found in Appendix I.

## II ANALYSIS OF SAMPLES

A total of 205 samples were analyzed on this contract. These included not only samples of quartz and quartz-growth related materials but also samples of Lithium Niobate, Indium, Phosphorous and Indium Phosphide. Almost all of the samples were analyzed using the analytical procedure developed in this laboratory. (See Appendix I) Some of the special samples, however, were analyzed by Emission Spectrogrpahy using a standard laboratory procedure.

The analytical procedure developed in this laboratory proved to be very good in that it allowed very little opportunity for sample contamination. The limiting factor in this procedure is not the dissolution of the sample but the final detection of the required elements. Atomic absorption was used during this contract. This limited our detection limits due to sample dilution. We intend to use an Inductively Coupled Plasma/Mass Spectrometer on these and similiar samples in the future. We expect this new state-of-the-art instrument to lower our detection limits for these elements to the parts-per-billion range.

Analytical results on all of these samples are reported in Tables I through Tables VII.

Table I
AF Contract F-19628-81-C-0072
Samples for Analysis

| No. | '81 Date | Identi. | µg/g Al | Fe | Na | Li | K | C | Ag | Pt | Ge |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. | 7/20 | QP-5 | 9. | 4. | 8. | 6. | 0.7 | < 40. | | < 1. | |
| 2. | 7/27 | X-23 | 453. | 10. | 11. | 126. | 8. | | | | |
| 3. | 8/24 | QA8-B | 1.3 | 1.7 | 6.3 | 0.8 | < 0.5 | | | | |
| 4. | 8/24 | QA8-T | 0.7 | 1.7 | 5.2 | < 0.5 | < 0.5 | | | | |
| 5. | 8/24 | X-33 | 6.9 | 2.7 | 9.4 | 1.7 | 0.7 | | | | |
| 6. | 9/29 | F-2 | 7. | 44. | 50. | 6. | 1. | < 40. | | | |
| 7. | 9/29 | QA-6 | 17. | 4. | 5. | 5. | < 0.5 | | | | |
| 8. | 9/29 | MOT | 16. | < 0.5 | 2. | 5. | < 0.5 | | | | |
| 9. | 9/29 | SAR | 28. | 0.5 | 14. | 3. | < 0.5 | | | | |
| 10. | 9/29 | Solution | 44. | | | | | | 4. | | |
| 11. | 10/21 | F-2 ReRun | 6. | 9. | 16. | 2. | < 0.5 | | | | |
| 12. | 10/21 | PQ-G-2 | 4. | 0.8 | 108. | 1. | < 0.5 | | | | |
| 13. | 10/21 | QA9-T | 2. | < 0.5 | 4. | < 0.5 | < 0.5 | | | | |
| 14. | 10/21 | QA9-B | 2. | 0.8 | 1. | 0.6 | < 0.5 | | | | |
| 15. | 10/21 | QA10-T | 3. | 0.9 | 0.5 | < 0.5 | < 0.5 | | | | |
| 16. | 10/21 | QA10-B | 2. | 1. | 4. | < 0.5 | < 0.5 | | | | |
| 17. | 10/21 | X-34 | 47. | 3. | 34. | 12. | < 0.5 | | | | |
| 18. | | QA-3 | 18. | 561. | 42. | | | | 18. | | |
| 19. | | X-12 | 96. | 444. | 122. | | | | 2.7 | | |
| 20. | | QP-1 | 22. | 2870. | 28. | 8. | 2.7 | | | < 10. | |
| 21. | | QP-2 | 8. | 708. | < 1. | 93. | 8.1 | | | < 10. | |
| 22. | | QA-5 | 13. | | 30. | < 1. | < 1. | | | | |
| 23. | | QA4-P | 15. | 2590. | 23. | 1.8 | 7.2 | | | | |
| 24. | | Sand | 7. | 330. | < 1. | 1. | 4. | | | | |
| 25. | | F-2 | 10. | 690. | 10. | 2.8 | 2.6 | | | | |
| 26. | | AMSR | < 2. | 420. | 10. | < 1. | 3.8 | | | | |
| 27. | | INC | 15. | 0.26 | 3.1 | 0.75 | 1.5 | | | | |
| 28. | | NC | 32. | 2240. | 17. | 4.3 | 4. | | | | |
| 29. | | NS | 55. | 1500. | 27. | 9.7 | 4. | | | | |
| 30. | | X-23 | 388. | 6.4 | 3.6 | 100. | < 1. | | | | |
| 31. | | QA-7 | 5.4 | 2.2 | 1. | 1.3 | < 1. | | | | |
| 32. | | X-32 | 68. | | | | | | | | |

Table I Continued

| No. | '82 Date | Identification | µg/g Al | Fe | Na | Li | K | C | Ag | Pt | Ge |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 33. | 2/9 | 1 | 9.8 | 2.2 | 20. | 0.8 | 3.1 | | | | |
| 34. | 2/9 | 2 | 7.9 | 1.7 | 32. | 0.5 | 5.7 | | | | |
| 35. | 2/9 | 3 | 27. | 4.1 | 69. | 1.4 | 10. | | | | |
| 36. | 2/9 | 4 | 33. | 3.0 | 24. | 2.7 | 6.5 | | | | |
| 37. | 2/26 | 12-B | 6. | 2.0 | 2.4 | 2.4 | 1. | | | | |
| 38. | 2/26 | 14-T | 5. | 27.0 | 0.6 | 11. | 0.5 | | | | |
| 39. | 2/2 | Metal Screw | 400. | major | 31. | | 17. | | | | |
| 40. | 2/2 | Metal Piece | 390. | major | 740. | | 110. | | | | |
| 41. | 5/11 | Never Seeze | 1-5% | | | | | | | | |
| 42. | 5/26 | QA16B | 9. | 0.9 | 28. | 3. | <0.5 | | <0.5 | | |
| 43. | 6/1 | QA18 | <0.5 | <0.5 | 0.5 | <0.5 | <0.5 | | | | |
| 44. | 8/30 | Mot. 1 | 21. | 2.7 | 10. | 2.5 | 3.4 | | | | |
| 45. | 8/30 | Mot. 2 | 6. | <0.5 | 8.3 | 1.3 | <0.5 | | | | |
| 46. | 8/30 | Mot. 3 | 3.9 | 2.5 | 890. | 2.8 | <0.5 | | | | |
| 47. | 8/30 | Mot. 4 | <0.5 | <0.5 | | | 1.6 | | | | |
| 48. | 8/30 | Mot. 5 | 2.1 | 5.8 | | | 4.6 | | | | |
| 49. | 8/30 | Mot. 6 | <0.5 | <0.5 | | | 0.7 | | | | |
| 50. | 8/30 | Mot. 7 | 2.3 | 3.1 | | | 3.6 | | | | |
| 51. | 8/30 | Mot. 8 | <0.5 | <0.5 | | | 3.7 | | | | |
| 52. | | Amethyst | 35. | 91. | 6.4 | 15. | 124. | | | | |
| 53. | | Citrine | 13. | 148. | 22. | 15. | 857. | | | | |
| 54. | | 14.31AOZ | 1.3 | <0.5 | 21. | 1.4 | <0.5 | | | | |
| 55. | | SARP 1 | 1.2 | 3.0 | 15. | 0.9 | <0.5 | | | | |
| 56. | | SARP 2 | 5.7 | 2.1 | 6.7 | 1.9 | <0.5 | | | | |
| 57. | | SARP 3 | 9.9 | 4.0 | 4.1 | 3.0 | <0.5 | | | | |
| 58. | | QA22X | 51. | 1.6 | 12. | 24. | <0.5 | | | | |
| 59. | | QA22Z | 35. | 1.2 | 11. | 17. | <0.5 | | | | |
| 60. | | 14.56-1 | 4.4 | 0.9 | 21. | 2.5 | <0.5 | Rerun of new sample | | | |
| 61. | 2/24/83 | Mot. 1 | 3.8 | 1.2 | 5.6 | 0.8 | <0.5 | | | | |
| 62. | 2/24/83 | Mot. 2 | 5.3 | 1.8 | 1.6 | 2.5 | 0.9 | | | | |
| 63. | 2/24/83 | Mot. 3 | 3.3 | 0.5 | 1.7 | 0.8 | <0.5 | | | | |

Table I Continued

| No. | Date | Identi. | µg/g Al | Fe | Na | Li | K | C | Ag | Pt | Ge |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 64. | 2/24/83 | Mot. 4 | 2.1 | 1.1 | 1.1 | 0.6 | < 0.5 | | | | |
| 65. | 2/24/83 | Mot. 5 | 0.6 | <0.5 | <0.5 | <0.5 | < 0.5 | | | | |
| 66. | 2/24/83 | Mot. 6 | 4.2 | 0.6 | 7.1 | 1.4 | < 0.5 | | | | |
| 67. | 2/24/83 | Mot. 7 | 23. | 9. | 11. | 6.9 | < 0.5 | | | | |
| 68. | 2/24/83 | Mot. 8 | 1.7 | 3.3 | 2.5 | 0.7 | < 0.5 | | | | |
| 69. | | GG-2-48 | 1.0 | 2.6 | 1.4 | <0.5 | < 0.5 | | | | |
| 70. | | Z-Growth | 7.5 | 1.0 | 2.2 | 2.0 | < 0.5 | | | | 598. |
| 71. | | QA21X | 5.3 | <0.5 | 0.9 | 1.5 | 0.5 | | | | |
| 72. | | QA21Z | 15. | <0.5 | 1.2 | 4.0 | < 0.5 | | | | |
| 73. | | QA20X | 28. | 0.5 | 3.3 | 7.6 | < 0.5 | | | | |
| 74. | | QA20Z | 17. | 0.8 | 1.1 | 4.7 | < 0.5 | | | | |
| 75. | | X39B | 6.5 | 3.2 | 2.1 | 1.5 | < 0.5 | | | | |
| 76. | | X39T | 3.7 | 1.4 | 1.2 | 0.9 | < 0.5 | | | | |
| 77. | | QA24Z | 3.8 | <0.5 | <0.5 | 1.0 | < 0.5 | | | | |
| 78. | | QA25X | 13. | <0.5 | 2.7 | 4.4 | < 0.5 | | | | |
| 79. | | X39X | 6.4 | 0.9 | 0.9 | 1.3 | < 0.5 | | | | |
| 80 | 4/12/83 | QA26BZ | 43. | .8 | 14. | 11. | 1.4 | | | | |
| 81 | " | X39Z | 3.6 | 2.7 | 1.5 | 1. | < .5 | | | | |
| 82 | " | X43BZ | 33. | 1.8 | 5.4 | 8.7 | 2.8 | | | | |
| 83 | " | X44 | 4.2 | .9 | 3.7 | 1.2 | < .5 | | | | |
| 84 | " | Z Mined Mat. | 28. | 1.7 | 2.0 | 7.5 | .8 | | | | |
| 85 | " | QP-10 | 9.7 | 5.6 | 2.2 | 2.6 | 1.1 | | | | |
| 86 | " | Material from Wall | 920. | 22.% | 6.6% | 127. | 32. | | | | |
| 87 | 5/16/83 | Z Seed FMQA-29 | 1.2 | 1.6 | 4.4 | 0.5 | 1.9 | | | | |
| 88 | " | FM Z Seed X37 | 3.3 | 1.4 | 7.8 | 0.9 | < 0.5 | | | | |
| 89 | " | Mot Z Mined Quartz | 3.1 | 3.7 | 7.7 | 1.2 | 9.6 | | | | |
| 90 | 5/31/83 | Z Mined Nutrient | 7.0 | <0.5 | 8.6 | 2.3 | 5.2 | | | | |
| 91 | 6/1/83 | No ID | 2.3 | <0.5 | 6.1 | 0.6 | 6.8 | | | | |
| 92 | " | Nutrient Z | 1.6 | <0.5 | 8.5 | 1.1 | 8.8 | | | | |

Table I Continued

| No. | Date | Identi. | μg/g Al | Fe | Na | Li | K | C | Ag | Pt | Ge |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 93. | 7/11/83 | QP-15 | 3.9 | 2.1 | 9.8 | 0.7 | 6.3 | | | | |
| 94. | " | X-35 | 3.6 | 2.5 | 18. | 1.5 | < 0.5 | | | | |
| 95. | 8/22/83 | Dynasil Glass Soaked | 4.5 | 2.5 | 2.8 | <0.5 | 3.7 | | | | |
| 96. | " | Dynasil Glass Untreated Clear | 3.3 | 2.3 | 2.1 | <0.5 | 1.2 | | | | |
| 97. | " | Dynasil Glass Untreated yellow | 1.0 | 1.0 | 1.7 | <0.5 | 1.7 | | | | |
| 98. | " | Dynasil Glass Cleaned | 1.0 | <0.5 | <0.5 | <0.5 | < 0.5 | | | | |
| 99. | " | X-47 Nutrient | 9.1 | <0.5 | 1.3 | 3.6 | 1.3 | | | | |
| 100. | " | Quartz no ident. | 4.0 | <0.5 | 1.3 | 1.2 | < 0.5 | | <0.5 | | |
| 101. | 9/6/83 | GC 6/21 | 8.3 | 1.3 | 4.7 | 2.1 | 1.7 | | | | |
| 102. | 9/9/83 | Soln. from 52ET11 | 2.2 | 0.4 | - | - | - | | | | |
| 103. | " | Sol.from QA-30 | 1.3 | 0.5 | - | - | - | | | | |
| 104. | " | QP-17 Potassium | 3.0 | 3.4 | 4.4 | 1.0 | 1.3 | | | | |
| 105. | 9/20/83 | GC11;T2-10 Mot. | 2.8 | 0.7 | 1.9 | 1.0 | <0.5 | | | | |
| 106. | " | GC11;T10-10 " | 2.7 | 0.5 | 3.2 | 0.9 | <0.5 | | | | |
| 107. | " | GC12;T1-2 " | <0.5 | <0.5 | 8.5 | 1.0 | 1.2 | | | | |
| 108. | " | GC12;T5-1 " | 1.8 | <0.5 | 8.0 | 1.1 | < 0.5 | | | | |
| 109. | " | GC13;T1-7 " | 2.6 | <0.5 | 2.8 | 0.9 | 2.5 | | | | |
| 110. | " | GC13;T3-1 " | 1.7 | <0.5 | 0.8 | 0.8 | < 0.5 | | | | |
| 111. | " | GC14;T5 " | 0.9 | 0.6 | 2.2 | <0.5 | 1.1 | | | | |
| 112. | " | GC15;T5 " | 0.5 | <0.5 | 1.5 | <0.5 | < 0.5 | | | | |
| 113. | " | GC16;T1-9 " | 3.4 | 0.6 | 6.4 | 1.0 | 0.8 | | | | |
| 114. | " | GC16;T2-9 " | 3.6 | <0.5 | 2.1 | 0.9 | < 0.5 | | | | |
| 115. | " | GC17;T2-7 " | 3.3 | <0.5 | 1.2 | 0.8 | 1.1 | | | | |
| 116. | " | GC17;T2-5 " | 4.1 | <0.5 | 1.3 | 1.3 | < 0.5 | | | | |
| 117. | " | GC18;T5 " | < 0.5 | <0.5 | 0.7 | <0.5 | 0.7 | | | | |
| 118. | 9/23/83 | Quartz 1 " | 3.2 | 1.1 | 396. | 0.8 | 0.5 | | | | |
| 119. | " | Quartz 2 " | 2.7 | 1.0 | 342. | 0.8 | 1.0 | | | | |
| 120. | " | Quartz 3 " | 1.1 | 1.1 | 287. | <0.5 | < 0.5 | | | | |

Table I Continued

| No. | Date | Identi. | µg/g Al | Fe | Na | Li | K | C | Ag | Pt | Ge |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 121. | " | Soln. 4 " | <0.5 | - | - | - | < 0.5 | | | | |
| 122. | 11/3/83 | Z Growth | 3.4 | <0.5 | 1.7 | 0.7 | < 0.5 | | | | 1100. |
| 123. | " | X-46A | 0.8 | 0.5 | 2.3 | <0.5 | < 0.5 | | | | < 10. |
| 124. | " | Comml. Seed Etched | 1.2 | 1.7 | 1.4 | <0.5 | 4.1 | | | | |
| 125. | " | SY2ET7 Seed Non Etch. | 5.1 | 2.1 | 9.9 | 1.8 | 6.0 | | | | |
| 126. | 11/2/83 | X49 X mined | 14.0 | 0.5 | 1.6 | 3.4 | 0.8 | | | | 2200. |
| 127. | " | X49 Z mined | 1.9 | <0.5 | 0.7 | <0.5 | < 0.5. | | | | 2170. |
| 128. | 11/28/83 | X53 low | 30.0 | 0.9 | 6.8 | 8.6 | 0.9 | | | | 3310. |
| 129. | " | X53 surface | 26.2 | 2.1 | 6.4 | 8.0 | < 0.5 | | | | 3820. |
| 130. | 12/6/83 | QP-16 Z mined | 3.4 | 0.5 | 5.6 | <0.5 | 5.0 | | | | |
| 131. | " | QP-18 Z mined | 1.1 | <0.5 | 1.4 | <0.5 | < 0.5 | | | | |
| 132. | 12/12/83 | QA31 X | <0.5 | 0.9 | <0.5 | <0.5 | < 0.5 | | | | |
| 133. | 12/12/83 | X48 | 1.4 | 1.8 | 1.0 | <0.5 | 0.5 | | | | |
| 134. | " | X50A | <0.5 | <0.5 | 1.9 | <0.5 | 2.2 | | | | |
| 135. | " | X50B | 0.9 | 0.8 | 1.5 | <0.5 | < 0.5 | | | | |
| 136. | " | X51X | 10.3 | 0.5 | 45. | 3.6 | 0.8 | | | | |
| 137. | 12/19/83 | 14.180-4 Sawyer | <0.5 | 0.5 | | | | | | | |
| 138. | " | 14.180-5 Sawyer | 0.7 | <0.5 | | | | | | | |
| 139. | " | 14.180-6 Sawyer | <0.5 | <0.5 | | | | | | | |

SPECIAL SAMPLES

| | | | Al | Fe | Na | Li | | | e | Mg | Ca | Cu |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 140. | 11/18/83 | QA30X X mined | 10.1 | <0.5 | 0.6 | 2.7 | < | < 0.5 | < .5 | < 0.5 | < 0.5 | 0.9 |
| 141. | " | QA30X Z mined | < 0.5 | < 0.5 | 0.5 | < 0.5 | <0.5 | <0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 |

TABLE II
Af Contract F-19628-81-C-0072
LITHIUM NIOBATE

µg/g

| Ident. | Si | Mg | Fe | Ni | Al |
|---|---|---|---|---|---|
| LNB-1 | 235. | 8. | 1360. | 320. | >2000. |
| 3LN-6 | 102. | >1000. | 1190. | 275. | >2000. |
| UCN-2-4 | 64. | 4. | 1030. | 230. | >2000. |

Table III
AF Contract F-19628-81-C-0072
Hi Purity Sodium Carbonate

| No. | '82 Date | Identi. | µg/g Al | Fe | Li | Mg | Ca |
|---|---|---|---|---|---|---|---|
| 1. | 3/15 | Unident. | 2. | 2. | 0.8 | | |
| 2. | 4/29 | Stauf. L | 4.4 | 0.5 | < 0.5 | 16. | 21. |
| 3. | 4/29 | Stauf. D | 2. | 0.9 | < 0.5 | 20. | 107. |
| 4. | 4/29 | Baker 126325 | < 0.5 | 0.5 | < 0.5 | 2. | 40. |
| 5. | 4/29 | Allied Gr 1285 | 4.8 | 2. | < 0.5 | 25. | 51. |
| 6. | 4/29 | Allied S 112 | < 0.5 | 5.1 | < 0.5 | 9. | 107. |
| 7. | 4/29 | CC #6 | 2.7 | < 0.5 | 0.6 | 16. | 92. |
| 8. | 4/29 | 0.83 Molar | 0.8 | < 0.5 | 280. | 1. | 3.3 |
| 9. | 5/5 | Stauf. L | 1.4 | | | | |
| 10. | 5/5 | Stauf. D | 0.8 | | | | |
| 11. | 5/5 | Baker 126325 | 0.6 | | | | |
| 12. | 5/5 | Allied Gr 1285 | 4.3 | | | | |
| 13. | 5/5 | Allied S 112(D) | 0.5 | | | | |
| 14. | 5/5 | CC #1382 | 2.0 | | | | |
| 15. | 5/5 | Allied S (109) | 1.7 | | | | |
| 16. | 5/5 | Baker 038104 | 0.5 | | | | |
| 17. | 5/5 | 0.81 Molar | < 0.5 | | | | |
| 18. | 5/5 | Mal. 7528 | < 0.5 | 0.9 | < 0.5 | 13. | 5.2 |

Table IV

AF Contract F-19628-81-C-0072

µg/g

| '82 Date | Identification | Al | Fe | Na | Li | K | Ca | Mg | Mn | P | Ti |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 11-3 | 103H | 46. | <0.5 | 3. | 9. | 1. | <0.5 | <0.5 | | | |
| " | 113T | 7. | <0.5 | 15. | 2. | 5. | <0.5 | <0.5 | | | |
| " | 015JB | 26. | <0.5 | <0.5 | 5. | 7. | <0.5 | <0.5 | | | |
| " | KAY | 17. | <0.5 | 11. | 0.6 | 2. | <0.5 | <0.5 | | | |
| " | COM | 51. | <0.5 | 5. | 3. | 6. | <0.5 | <0.5 | | | |
| " | E8-51 | 19. | 8. | 625. | 7. | <0.5 | <0.5 | <0.5 | | | |
| " | E8-51 Supply After | 32. | 15. | 1240. | 3. | <0.5 | <0.5 | <0.5 | | | |
| " | E8-51 " " Before | 14. | 1. | 3. | 0.5 | <0.5 | <0.5 | <0.5 | | | |
| " | 14.5 Supply After | 23. | 6. | 1890. | 3. | <0.5 | <0.5 | <0.5 | | | |
| " | 13.191 " " Before | 6. | <0.5 | 0.5 | 0.5 | <0.5 | <0.5 | <0.5 | | | |
| 12-16 | 14.53-1 | 0.9 | <0.5 | 1.4 | <0.5 | <0.5 | 1.8 | 0.6 | | | |
| " | 14-53-2 | 1.1 | <0.5 | 0.8 | 0.5 | <0.5 | <0.5 | <0.5 | | | |
| " | 14-53-3 | 7.0 | <0.5 | 23. | 2.2 | <0.5 | <0.5 | <0.5 | | | |
| " | 14-53-4 | 1.0 | <0.5 | 0.6 | <0.5 | <0.5 | <0.5 | <0.5 | | | |
| " | 14-56-1 | 3.0 | 33. | 22. | 2.3 | <0.5 | <0.5 | <0.5 | | | |
| " | 14-56-2 | 27. | <0.5 | 25. | 7.3 | <0.5 | <0.5 | <0.5 | | | |
| " | 14-56-3 | 12. | 0.7 | 4.9 | 3.6 | <0.5 | <0.5 | <0.5 | | | |
| " | F39B | 880. | - | - | - | - | 730. | 919. | 213. | <100. | 760. |
| " | F40G | 400. | - | - | - | - | 0.5 | 203. | 37. | <100. | 78. |
| " | J15G | 787. | - | - | - | - | 3.5% | 1.3% | 1.3% | 220. | 278. |

Table V

AF Contract F-19628-81-C-0072

µg/g

| Sample | Al | Ca | Mg | Mn | P | Ti | In | Ga |
|---|---|---|---|---|---|---|---|---|
| F26 Bottom | 780. | 10800. | 2020. | 1160. | 100. | 690. | | |
| H38 Upper Wall | 2590. | 1480. | 1990. | 1650. | 100. | 505. | | |
| In-Ga Mix | | | | | | | 94.7% | 5.2% |

TABLE VI

Indium Phosphide Samples

µg/g

| Sample | Fe | Ge | Sn |
|---|---|---|---|
| 8-29-83 | | | |
| 1 | ND | ND | 35. |
| 2 | ND | ND | 53. |
| 3 | ND | ND | 63. |
| 4 | ND | ND | 50. |
| 5 | ND | ND | 61. |
| 6 | ND | ND | 76. |
| 7 | ND | ND | 640. |
| 8 | ND | ND | 7800. |
| A | ND | 2500. | ND |
| B | ND | 2000. | ND |
| C | ND | 126. | ND |
| D | 580. | 50. | ND |
| 9-20-83 | | | |
| Special (Ahern) | 2.8 | | |

Table VII
AF Contract F-19628-81-C-0072
Indium and Phosphorous Samples

| Sample Identification | μg/g Al | Fe | Zn | Sn | Ga | Mg |
|---|---|---|---|---|---|---|
| Indium Corp. Indium | <1. | <1. | <0.5 | <2. | <30. | <0.5 |
| MPC Indium | <1. | <1. | <0.5 | <2. | <30. | <0.5 |
| MPC Red P form II | 34. | <1. | <0.5 | <2. | <30. | <0.5 |
| MPC Red P (used) | <1. | <1. | <0.5 | <2. | <30. | <0.5 |
| Red P form II fresh | 38. | 17. | 1.0 | 3. | <30. | <0.5 |
| Red P form II spent | 110. | 1090. | 4. | 31. | <30. | 1.8 |
| InP LV-90 | <1. | 2. | <0.5 | 26. | <30. | <0.5 |

Appendix I

## Analysis of Quartz for Impurities

Application: This method is applicable to the determination of aluminum, iron, sodium, lithium, potassium and silver. Two grams of sample are required for duplicate analyses.

Summary: A sample is dissolved in teflon in a Parr bomb using ultra-pure HF acid. The silica in the sample is then volatilized in platinum. The impurity containing residue is put into solution using ultra-pure $HNO_3$ acid. The analysis is completed using Atomic Absorption.

Apparatus:

1. Perkin-Elmer 5000 AA with graphite furnace capability.
2. Parr 4745 acid digestion bomb.
3. Platinum crucibles.
4. Volumetric flasks-10 ml.
5. Furnace or oven capable of 130 C.

Reagents and Standards:

1. Ultra-pure HF acid.
2. Ultra-pure $HNO_3$ acid.
3. Ultra-pure $H_2SO_4$ acid.
4. Ultra-pure acetone.
5. Demineralized water.
6. AA standards for the desired elements.

Analysis:

1. The quartz ingot is washed well with acetone and water to remove any sealing wax used in sawing.

2. The ingot is then wrapped in several layers of kleenex and broken into shards with a hammer.

3. The shards are then collected in a cleaned pyrex beaker and washed repeatedly with acetone, HCl, $HNO_3$ and demineralized water. They are finally washed with several changes of demineralized water. The shards are then dried carefully.

4. A sample is weighed from these cleaned and dried shards and placed in a similarily cleaned teflon cup for the Parr 4745 acid digestion bomb. The shards should be small.

5. Five ml. (for a 1 gm sample) of ultra-pure HF is added to the teflon cup and the bomb is carefully assembled.

6. The bomb is then placed in an oven and heated at 130 C overnight.. This should completely dissolve the sample but additional time may be needed if the shards are very large.

7. The bomb is then removed from the oven and allowed to cool.

8. The bomb is then carefully opened (wear rubber gloves and safety glasses) and the teflon cup removed. The cup is opened carefully to avoid spilling the contents and washed carefully into a clean platinum crucible.

9. Add 2 drops of ultra-pure $H_2SO_4$ and carefully heat the crucible to evaporate the HF and thus volatilized the silica as silicon tetrafluoride.

10. The residue remaining in the crucible (containing the impurity elements) is put back into solution by adding 3 drops of ultra-pure $HNO_3$ and a small amount of demineralized water. Heat gently but do not evaporate.

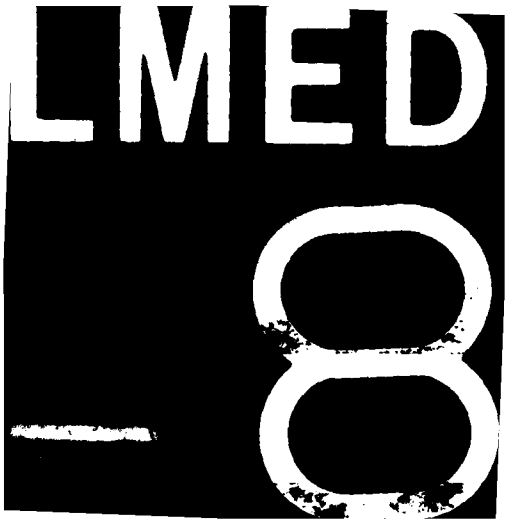
11. The contents of the crucible are very carefully transferred to a cleaned 10 ml volumetric flask and made to volume with demineralized water.

12. The analysis is completed using Atomic Absorption and the graphite furnace if necessary. Use carefully prepared standards and blanks.

    Note: All glassware must be carefully cleaned before use. Do not get anything from the outside of the crucibles in the solution. Use rubber or plastic gloves and eye shields when handling HF and other acids.

# *MISSION*
# *of*
# *Rome Air Development Center*

*RADC plans and executes research, development, test and selected acquisition programs in support of Command, Control Communications and Intelligence ($C^3I$) activities. Technical and engineering support within areas of technical competence is provided to ESD Program Offices (POs) and other ESD elements. The principal technical mission areas are communications, electromagnetic guidance and control, surveillance of ground and aerospace objects, intelligence data collection and handling, information system technology, ionospheric propagation, solid state sciences, microwave physics and electronic reliability, maintainability and compatibility.*


LMED
8